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भारतीय मानक

पॉलीइथाइलीन ग्लाइकोल 200 — विशिष्टि

Indian Standard

POLYETHYLENE GLYCOL 200 — SPECIFICATION

UDC 547·422

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

April 1992

Price Group 3

Organic Chemicals (Miscellaneous) Sectional Committee, PCD 9

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Polyethylene glycols are a series of polymers which exhibit good lubricity, heat stability and inertness to many chemicals. These are represented by the molecular formula $H(OCH_2 \cdot CH_2)_nOH$, where the numerical suffix is an indication of the molecular mass, the changes in which are reflected in their physical properties. The series of molecular mass of the product may lie in the range of 200 to 1 500. In general, with the increase in molecular mass, the polyethylene glycols show a decrease in water solubility, vapour pressure, hygroscopicity and solubility in organic solvents, whereas they show an increase in melting point, specific gravity, flash point and viscosity.

Polyethylene glycols are used in the preparation of ointment bases replacing advantageously traditional materials like paraffin and petroleum products. They are also extensively used in the preparation of cleansing and vanishing creams, in hair-dressing preparations and certain types of antiseptic tooth pastes. The liquid polyethylene glycols are exceptionally useful solvents, binders and plasticizers in the production of corks. Polyethylene glycols are extensively used in the preparations of textile auxiliaries, mainly in the form of esters. For the preparation of esters, lauric, stearic and oleic acids may be esterified with polyethylene glycols 200, 400, 600 and 1 000. The mono esters of lauric, stearic and oleic acids with polyethylene glycols are used in the preparation of emulsions for scouring and rinsing textiles where it exerts a dispersing action on lime and magnesium insoluble soaps caused by hard water, the emulsions in drying oil are used in the sizing of rayon crepe and for starch and other mucilage sizing of textiles.

Polyethylene glycols are also used as water soluble non-volatile lubricants for moulds in the rubber industry; liquid polyethylene glycols are also used as lubricants for type air-bags in the manufacture of tyres.

Polyethylene glycol 200 is an important ingredient in the manufacture of heavy duty hydraulic brake fluids.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

POLYETHYLENE GLYCOL 200 — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for polyethylene glycol 200.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	*Title*
265 : 1987	Hydrochloric acid (*third revision*)
915 : 1975	One-mark volumetric flasks (*first revision*)
1070 : 1977	Water for general laboratory use (*second revision*)
4161 : 1967	Nessler cylinders
4905 : 1968	Methods for random sampling

3 REQUIREMENTS

Description

The material at room temperature shall be colourless, odourless liquid and shall be soluble in water as well as in organic solvents, such as alcohols, glycol ethers, ketones and aromatic hydrocarbons. The material shall also comply with the requirements given in Table 1 when tested according to the methods given in Annex A with reference to the relevant clauses of Annex A as given in col 4 of Table 1.

4 PRECAUTIONS IN HANDLING

Polyethylene glycol 200 is non-volatile and posses-ses a very low order of toxicity and having insignificant effect on the skin. As such no special precautions are necessary in handling. However, handling or storing polyethylene glycol 200 at elevated temperature over prolonged periods is recommended to be carried out under a nitrogen blanket. Handling at temperature in excess of 140°C is not recommended.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in lacquer-lined mild steel drums.

Table 1 Requirements for Polyethylene Glycol 200

(*Clause* 3.1)

Sl No.	**Characteristic**	**Requirement**	**Method of Test (Ref to Cl No. in Annex A)**
(1)	(2)	(3)	(2)
i)	Colour, Hazen units, *Max*	15	A-2
ii)	Acidity (as acetic acid), percent by mass, *Max*	0·05	A-3
iii)	Viscosity at 99°C, centistokes	4·0 to 4·2	A-4
iv)	Average molecular mass	190 to 210	A-5
v)	*p*H of 5 percent (*m*/*v*), aqueous solution	4·0 to 7·0	A-6

5.2 Marking

The containers shall be marked with the following information:

a) Name of the material;
b) Indication of the source of manufacture;
c) Net mass of the material in the container; and
d) Lot or batch number, in code or otherwise.

5.2.1 The container may also be marked with the Standard Mark.

6 SAMPLING

Representative samples of the material shall be drawn and their conformity to the standard determined as prescribed in Annex B.

ANNEX A

(*Clause* 3.1 and *Table* 1)

METHODS OF TEST FOR POLYETHYLENE GLYCOL 200

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis

A-2 DETERMINATION OF COLOUR

A-2.0 Outline of the Method

The colour of the material is compared with that of the colour standard and expressed in terms of Hazen colour units. One Hazen colour unit is defined as the colour of an aqueous solution containing 1 part per million of platinum in the form of chloroplatinic acid and 2 parts per million of cobaltous chloride ($CoCl_2.6H_2O$).

A-2.1 Apparatus

A-2.1.1 *Nessler Cylinders*

Two 100-ml (*see* IS 4161 : 1967).

A-2.1.2 *One-Mark Volumetric Flasks*

250-ml and 500-ml capacities (*see* IS 915 : 1975).

A-2.2 Reagents

A-2.2.1 *Cobaltous Chloride Hexahydrate*

Solid

A-2.2.2 *Hydrochloric Acid*

(Conforming to IS 265 : 1987).

A-2.2.3 *Chloroplatinic Acid*

Dissolve 250 mg of platinum in small quantity of aquaregia contained in a glass or porcelain basin by heating on a water-bath. When the metal has dissolved, evaporate the solution to dryness. Add 1 ml of hydrochloric acid and again evaporate to dryness. Repeat this operation twice.

A-2.3 Preparation of Colour Standard

Dissolve 0·50 g of cobaltous chloride hexahydrate and whole of the chloroplatinic acid in 50 ml of hydrochloric acid. Warm, if necessary, to obtain a clear solution and after cooling, pour into the 500-ml one-mark volumetric flask. Dilute with water to the mark.

A-2.3.1 Pipette 7·5 ml of the solution into a 250-ml one-mark volumetric flask and dilute with water to the mark. The diluted solution corresponds to a colour of 15 Hazen units and should always be freshly prepared.

A-2.4 Procedure

Fill one of the Nessler cylinders to the mark with the material to be tested, and the other with the colour standard. Compare the colours using a white background. The material shall be taken to have passed the test if the colour of the sample is not darker than that of the colour standard.

A-3 DETERMINATION OF ACIDITY (AS ACETIC ACID)

A-3.1 Procedure

Weigh about 50 g of sample to the nearest 0·01 g in one litre conical flask. Add 500 ml of distilled water to the above flask, and swirl the flask gently so that uniform mixing of the sample and water takes place. Titrate the contents of the flask with 0·1 N sodium hydroxide using phenolphthalein as indicator. Carry out a blank determination in exactly the same way as above but without the sample.

A-3.2 Calculation

$$\text{Acidity (as acetic acid)} = \frac{(V_1 - V_2) \times N \times 6}{M}$$

where

V_1 = volume of sodium hydroxide solution in ml, used for sample titration;

V_2 = volume of sodium hydroxide solution in ml, used for blank;

N = normality of sodium hydroxide; and

M = mass in g of the sample for test.

A-4 DETERMINATION OF VISCOSITY

A-4.1 Apparatus

A-4.1.1 *Bath with Thermostat*

Any transparent liquid bath, provided that it is of sufficient depth so that at any time during the measurement no portion of the sample in the viscometer shall be less than 2 cm below the surface of the bath liquid or less than 2 cm above the bottom of the bath.

This bath shall have thermostatic temperature control arrangement and the set temperature shall not vary by more than ±0·1°C.

A-4.1.2 *Thermometer*

Calibrated and capable of reading to 0·01°C accuracy.

A-4.1.3 *Viscometer*

Glass capillary type, calibrated which will give a flow time of at least 200 seconds with the sample to be tested.

A-4.1.4 *Viscometer Holder*

To enable the viscometer tube to be suspended in the bath in vertical position.

A-4.1.5 *Stop Watch*

To read the flow time of the sample accurately up to 0·2 seconds.

A-4.2 Procedure

Fill the bath with monoethylene glycol and maintain the temperature of the bath exactly at 99°C with the help of thermostatic control. In order to obtain the most reliable temperature measurement it is recommended that two thermometers with valid calibration certificate be used.

Charge viscometer with the sample and allow the charged viscometer to remain in the bath long enough to reach the test temperature. Use suction to adjust the head level of the test sample to a position in the capillary arm about 5 mm ahead of the first timing mark. With the sample flowing freely, measure in seconds to within 0·2 seconds the time required for the meniscus to pass from the first timing mark to the second.

A-4.2.1 Repeat the measurement. The timings should agree within 0·2 percent. Use the average timing for calculations.

A-4.3 Calculation

Calculate the viscosity ρ from the average measured flow time t and the calibration constant of the viscometer by means of the following equation:

$$\text{Kinematic viscosity, } \rho = Ct$$

where

ρ = kinematic viscosity, centistokes;

C = calibration constant of the viscometer, centistokes per second; and

t = flow time, seconds.

A-5 DETERMINATION OF AVERAGE MOLECULAR MASS

A-5.0 Methods

Two methods, namely, Method A and Method B have been prescribed.

A-5.1 Method A

A-5.1.0 *Outline of the Method*

The sample is esterified by refluxing with an excess of phthalic anhydride reagent in pyridine. The excess reagent is then hydrolyzed with water and the liberated acid is titrated to the phenolphthalein end point. The difference between this titration and that of a blank, in which the same quantity of reagent is hydrolyzed with water in the absence of glycol, gives after correction for the acidity of the sample, a measure of the hydroxyl groups present in the sample. The apparent molecular mass of the sample is then derived from the amount of acid bound by the hydroxyl groups, the number of hydroxyl groups per molecule and the mass of sample used.

A-5.1.1 *Reagents*

A-5.1.1.1 *Phthalic anhydride reagent*

Dissolve 42 g of phthalic anhydride of at least 99 percent purity in 300 ml of dry pyridine. Allow to stand overnight and store in a brown bottle. Prepare fresh reagent each week.

A-5.1.1.2 *Sodium hydroxide solution*

Dissolve 45 g of sodium hydroxide in 45 ml of freshly boiled and cooled distilled water. Allow any precipitate to settle, filter through a plug of glasswool and dilute with CO_2 free distilled water to 1 litre. Standardize against potassium hydrogen phthalate using phenolphthalein as an indicator.

A-5.1.1.3 *Phenolphthalein indicator solution*

1 percent in pyridine.

A-5.1.2 *Procedure*

Weigh into a 250-ml conical flask with ground glass joint, to the nearest 0·1 mg, such an amount of sample (1/200 of the expected molecular mass) that approximately one-third of the available phthalic anhydride is consumed. Add by means of volumetric pipette 25 ml of phthalic anhydride reagent and attach the flask to a dry reflux condenser by means of a ground glass joint moistened with pyridine and vigorously reflux on a hot plate for 30 minutes.

Remove flask and condenser from the hot plate and allow to stand for 15 minutes. Wash down the condenser first with 25 ml of pyridine and then with 25 ml of water. Remove the flask from the condenser. Add 10 drops of phenolphthalein indicator solution in pyridine to the flask and titrate with standard sodium hydroxide solution to the first faint pink colour which persists for 15 seconds.

A-5.1.2.1 Carry out a blank determination without using sample following the procedure given in **A-5.1.2.**

A-5.1.2.2 Determine the acidity of sample according to the method in **A-3.**

A-5.1.3 *Calculation*

$$\text{Molecular mass} = \frac{M \times M_1 \times 2000}{M_1 (B - A) N + (M \times C \times N_1)}$$

where

A = volume of sodium hydroxide solution used for titration of the sample solution;

B = volume of sodium hydroxide solution used for the blank titration;

C = volume of sodium hydroxide solution used for the acidity titration;

N = normality of the sodium hydroxide solution used for titration;

N_1 = normality of the sodium hydroxide solution used for determination of acidity;

M = mass of sample, g; and

M_1 = mass in g of sample used for determination of acidity.

A-5.2 Method B

A-5.2.1 *Apparatus*

A-5.2.1.1 *Erlenmeyer flasks*

250-ml standard ground glass stoppered.

A-5.2.1.2 *Pipette*

A-5.2.1.3 *Burette*

A-5.2.1.4 *Reflux condensers*

Standard ground glass joints.

A-5.2.1.5 *Steam bath*

A-5.2.2 *Reagents*

A-5.2.2.1 *Pyridine*

A. R. grade

A-5.2.2.2 *Acetic anhydride*

A-5.2.2.3 *Pyridine-acetic anhydride reagent*

Freshly prepared before use by mixing 3 volumes of pyridine with 1 volume of acetic anhydride.

A-5.2.2.4 *Normal butyl alcohol*

Neutralized with 0·5 N ethanolic KOH to faint pink phenolphthalein end point.

A-5.2.2.5 *Ethanolic potassium hydroxide solution*

0·5 N, standardized.

A-5.2.2.6 *Phenolphthalein indicator solution*

1 percent in ethanol.

A-5.2.3 *Procedure*

A-5.2.3.1 Weigh to the nearest 0·1 g of the sample into the Erlenmeyer flask.

A.5.2.3.2 Pipette 5·0 ml of the pyridine-acetic anhydride reagent into the flask containing the sample.

A-5.2.3.3 Thoroughly mix the contents by gentle swirling.

A-5.2.3.4 Pipette 5·0 ml of pyridine-acetic anhydride reagent into an empty flask for the reagent blank.

A-5.2.3.5 Place the flasks on steam bath under reflux condensers and heat for one hour.

A-5.2.3.6 Add 10 ml of water through the condenser to the flask. Heat on the steam bath for 10 min with reflux condensers attached.

A-5.2.3.7 Allow the flasks to cool to room temperature with condensers attached.

A-5.2.3.8 Add 25 ml of neutralized butyl alcohol about half through the condensers, remove condenser and use remaining butyl alcohol to wash down the sides of flasks.

A-5.2.3.9 Add 1 ml of phenolphthalein indicator solution. Titrate to a faint pink end point with 0 5 N alcoholic KOH solution.

A-5.2.3.10 Weigh accurately about 10 g of the sample into another flask for determination of acidity.

A-5.2.4 *Calculation*

$$\text{Molecular mass} = \frac{2\,000 \times W}{\left[B + \frac{(WA)}{C} - S \right] \times N}$$

where

A = ml of KOH solution required for acidity titration,

B = ml KOH solution required for blank,

C = mass in g of the sample used for acid value acidity titration,

S = ml of KOH solution required for titration of acetylated sample, and

W = mass in g of sample used for acetylation.

A-6 DETERMINATION OF *p*H

A-6.1 Procedure

Weigh 10 ± 0·1 g of sample in a clean 500-ml glass beaker. Add 190 ml of distilled water (*p*H 7·0 ± 0·1) and mix the contents till the solution is complete. Measure *p*H using *p*H meter which has been calibrated with glass and calomel electrodes. Report the *p*H to the nearest 0·1 unit.

ANNEX B

(*Clause* 6)

SAMPLING FOR POLYETHYLENE GLYCOL 200

B-1 GENERAL REQUIREMENTS

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 The sampling instrument shall be clean and dry and shall be made of low or reduced spark generating material.

B-1.3 The samples, the material being sampled, the sampling instrument and the containers for samples shall be protected from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking or stirring or both, or by rolling, so as to bring all portions into uniform distribution.

B-1.5 The samples shall be placed in suitable, clean, dry and airtight glass containers preferably of amber or blue colour.

B-1.6 The sample containers shall be of such a size that they are almost, but not completely, filled by the sample.

B-1.7 Rubber stoppers or composition corks shall not be used for closing the sample bottles.

B-1.8 Sealing wax or other plastic material, if used, shall be applied in such a way that it does not contaminate the sample when the bottles are opened.

B-1.9 Each sample container shall be protected by covers of oilproof paper, metal foil, viscose or other suitable impervious material over the stopper to keep away moisture and dust from the mouth of the bottle and to protect it while being handled.

B-1.10 Each sample container shall be sealed airtight with a suitable stopper after filling and marked with full details of sampling, such as the date of sampling, the year of manufacture of the material, the batch number, the name of the sampler, etc. Particular care shall be taken to ensure that sealing methods do not contaminate the sample.

B-2 SAMPLING INSTRUMENT

B-2.0 The following forms of sampling instruments may be used:

a) Sampling bottle or can, for taking samples from various depths of large tanks; and

b) Sampling tube.

B-2.1 Sampling Bottle or Can

It consists of weighted bottle or metal container with removable stopper or top, to which is attached a light chain (*see* Fig. 1). The bottle or can is fastened to a suitable pole. For taking a sample, it is lowered in the tank to the required depth, and the stopper or top is removed by means of the chain for filling the container.

B-2.2 Sampling Tube

It is made of metal or thick glass and is about 20 to 40 mm in diameter and 400 to 800 mm in length (*see* Fig. 2). The ends are conical and reach 5 to 10 mm diameter at the tips. Handling is facilitated by two rings at the upper end. For taking a sample, the apparatus is first closed at the top with the thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

B-2.2.1 For small containers, the size of the sampling tube may be altered suitably.

B-3 SCALE OF SAMPLING

B-3.1 Lot

In any consignment, all the containers of the same size and drawn from same batch of manufacture shall consitute a lot.

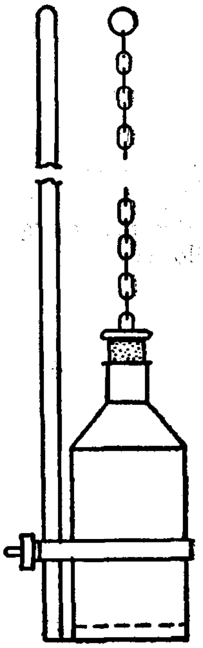

FIG. 1 SAMPLING BOTTLE OR CAN

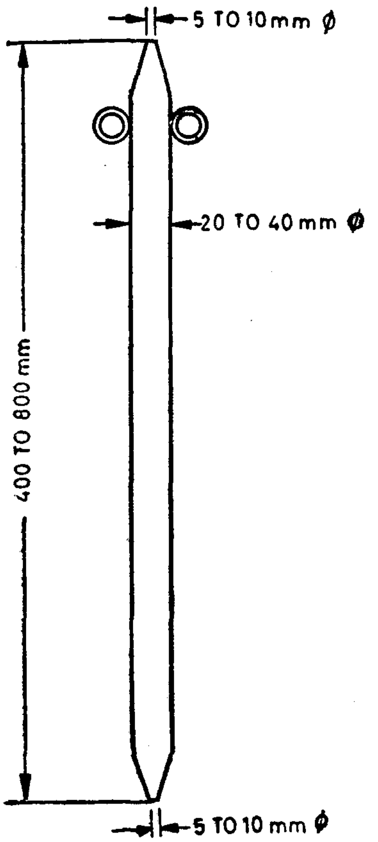

FIG. 2 SAMPLING TUBE

B-3.2 Tests shall be conducted on each lot separately for ascertaining its conformity to the requirements of this specification. The number of containers to be chosen at random from the lot for this purpose shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table 2.

B-3.3 The containers shall be selected from the lot at random and in order to ensure the randomness of selection, the method given in IS 4905 : 1968, may be followed.

Table 2 Scale of Sampling
(*Clause* B-3.2)

Number of Containers in the Lot	Number of Containers to be Selected
(1)	(2)
Up to 25	3
26 ,, 100	4
101 ,, 150	5
151 and above	7

B-4 PREPARATION OF TEST SAMPLES

B-4.1 From each of the containers selected according to **B-3.2** and **B-3.3**, small portions of the material shall be drawn with the help of the sampling instrument (*see* **B-2**) from several positions at the upper, middle and bottom layers. These portions shall be mixed together so as to yield a quantity of material not less than 1 000 ml representative of that container.

B-4.2 From each of these individual samples, an equal quantity of material shall be taken and mixed thoroughly to form a composite sample, of about 1 000 ml. The composite test sample shall be divided into three equal parts, one for the purchaser,, another for the supplier and the third to be used as a referee sample.

B-4.3 The parts of the composite test sample, obtained according to **B-4.2**, shall be transferred to separate containers and shall be sealed and marked with full identification particulars given in **B-1.10**.

B-4.4 The referee test sample shall bear the seals of both the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier to be used in the case of any dispute between the two.

B-5 CRITERIA FOR CONFORMITY

The lot shall be declared as conforming to the requirements of this specification, if the composite sample does not fail to satisfy any of the specified requirements.

Standard Mark

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